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CHEMICAL SYSTEMS LABORATORY TECHNICAL REPORT

ARCSL-TR-81064

EVALUATION OF REPLACEMENT RED SMOKE DYES  
FOR 1-N METHYLAMINOANTHRAQUINONE

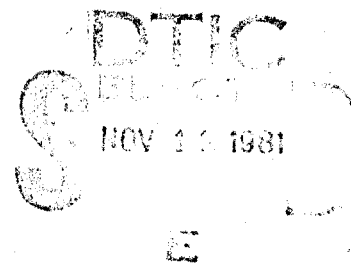
by

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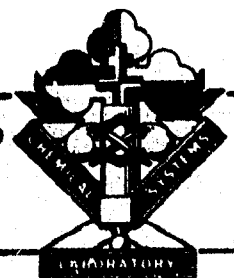


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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AKCSL-TR-81054	2. GOVT ACCESSION NO. AD-A107 286	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) EVALUATION OF REPLACEMENT RED SMOKE DYES FOR 1-N METHYLAMINOANTHRAQUINONE.		5. TYPE OF REPORT & PERIOD COVERED Technical Report November 1979 - May 1980
7. AUTHOR(s) Ellen S. Vigus Albert Deiner		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Commander/Director, Chemical Systems Laboratory ATTN: DRDAR-CLN-D Aberdeen Proving Ground, Maryland 21010		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Project 1A98772
11. CONTROLLING OFFICE NAME AND ADDRESS Commander/Director, Chemical Systems Laboratory ATTN: DRDAR-CLJ-R Aberdeen Proving Ground, Maryland 21010		12. REPORT DATE October 1981
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) 12 17		13. NUMBER OF PAGES 20
		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE NA
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Red smoke dyes 1-N Methylaminoanthraquinone 9-Diethylaminorosindone O-Methoxyphenylazo- $\beta$ -naphthol Pyrotechnics Sudan Red G M-18 grenade Toxicology FDA dyes		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A list of 114 dyes was compiled from a literature search for replacement red smoke dyes for 1-N methylaminoanthraquinone. Two of the dyes reviewed (9-diethylaminorosindone and O-methoxyphenylazo-B-naphthol) were found to meet the requirements for use in pyrotechnics. There are insufficient toxicological data at this time to assess the health effects of these dyes.		

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## PREFACE

The work described in this report was authorized under Project 1A98772, Engineering Study to Determine Feasibility of Replacing Dye, Disperse Red 9. This work was started in November 1979 and completed in May 1980.

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## EVALUATION OF REPLACEMENT RED SMOKE DYES FOR 1-N METHYLAMINOANTHRAQUINONE

### 1. INTRODUCTION

The objective of this work was to compile a list of possible replacement compounds for the dye currently employed to generate red smoke by pyrotechnic methods. The red dye now used is 1-N methylaminoanthraquinone.

### 2. METHOD

A list of dyes used or tested for use in pyrotechnics was compiled by reviewing open literature and reports available in the Technical Library of Chemical Systems Laboratory.

Representatives from dye manufacturing companies were consulted to determine whether any new available dyes could be employed for use with pyrotechnic compositions.

The list of Food and Drug Administration dyes subject to certification (revised April 1, 1979) was reviewed for replacement dyes. These dyes are commercially available, and toxicological testing has already been performed on each.

### 3. RESULTS

Table 1 lists the dyes from the open literature and from reports in the Technical Library which have been used or tested for use in pyrotechnic smoke compositions.

Included with the list of dyes is the following:

- a. Manufacturer, if available.
- b. Other names - commercial or chemical.
- c. Colour Index (CI) number (Numbers before 10000 are no longer listed in the current Colour Index.)

Table 2 lists the Food and Drug Administration dyes reviewed. Current Colour Index numbers are noted.

Consultations with dye manufacturers yielded one possible dye, Rhodamine B.

Table 1. Dyes Used or Tested for use in Pyrotechnics

Dye		Reference
1 Acetazuril Scarlet B	Made by DuPont	1, 3
2 AD 687		3
3. AD 722		3
4. AD 779		2
5. Alpha-amino anthraquinone		1, 4, 5
6 Amino azobenzene		3
7 Amino azotoluene- $\beta$ -naphthol		
8 2-Amino biphenyl- $\beta$ -naphthol		
9 1-Amino-4-hydroxy anthraquinone		1, 3, 4
10. Aniline-1-hydroxy naphthalene-4-sulfonic acid	Celanthrene Red 3B Made by DuPont	3
11. Aniline azo- $\alpha$ -naphthol		6
12. 5-Anilinobenzo (a) phenazine		1
13 o-Anisidine-1-hydroxy naphthalene-4-sulfonic acid		3
14. p-Anisidine-1-hydroxy naphthalene-4-sulfonic acid		3
15 o-Anisidine azo $\alpha$ -naphthol		6
16 o-Anisidine azo resorcinol		6
17. 4-Anisole azo $\beta$ -naphthol		2
18 Azo Red K 1858		7
19 1-(4-Benzamido-2,5-dimethoxy phenylazo)-2-naphthol	Rosinduline GX, CI 828	1
20. 1-(2-Biphenylazo)-2-naphthol		1
21. Brilliant Red R		7
22. 5-Bromo-2(5,7-dibromo-3-oxo-3(2)-indolydine)-3(2)-thianaphthenone		1
		CI 73620 (Vat Dye) <sup>8</sup>
23 Calcolake Red D		7
24 p-Chloroaniline- $\beta$ -naphthol		1, 3
25. p-Chloro-o-anisidine-1-hydroxynaphthalene-4-sulfonic acid		3
26 4-Chlor-2-anisole-azo- $\beta$ -naphthol		2, 3
27 4-Chlor-2,5-dimethoxybenzene-azo- $\beta$ -naphthol		1, 2, 3
28. 5-Chloro-2(hydroxy-1-naphthylazo)-p-toluene sulfonic acid	Calcolake Red C Sulfanthrene Red 3B CI 1212	1
29 5,5'-Chloro-6,6'-methylthioindigo		1
30. 4-(p-Chlorophenylazo)-N,N-dihydroxy ethylaniline		1
31. 4-(p-Chlorophenylazo)-N-ethyl-N-(2-hydroxyethyl) aniline		1
32 Cresidine-1-hydroxy naphthalene-4-sulfonic acid		3
33 1,5-Diamino anthraquinone		4
34. 1,4-Dibenzoylamino anthraquinone		1
35. 2,4-Dichloroaniline- $\beta$ -naphthol		4
36. 2,5-Dichloroaniline- $\beta$ -naphthol		1, 3
37 2,5-Diethoxyaniline- $\beta$ -naphthol		1, 3
38. 9-Diethylamino rosindone	9-Diethylamino-7-phenyl-5-benzo (a) phenazinone (MIL-D-3614) Rosindone Fractional AD *CI 12156 (Solvent Red 80) <sup>8</sup>	1, 2, 5, 7 9-11
39 9-Diethylamino-7-p-tolyl-5-benzo (a)-phenazinone		1, 7, 9
40. N,N-Diethyl-p,p'-nitrophenylazoaniline		1
41 2,5-Dimethoxybenzene-azo- $\beta$ -naphthol		1, 2, 3
42. 1,5-Dimethylamino anthraquinone		6
43. 1,8-Dimethyl-3,6-dichloro thioindigo		3
44. 4-(2,4-Dinitro phenylazo) phenol		1
45. 6-Ethoxy-2-amino-benzthiazole-ethylanilol		1, 3
46. 5-Ethyl-2-anisole-azo- $\beta$ -naphthol		2
47. Federal Continental Red		3

<sup>8</sup> See footnote at end of table.

Table 1. (Contd)

Dye		Reference
48 Federal Independence Red	*CI 12120 (Pigment Red 3) <sup>8</sup>	3
49 Federal Lincoln Red	Chlorinated p-nitro phenylazo-2-naphthol *CI 12085 (Pigment Red 4) <sup>8</sup>	1, 3
50 Federal Signal Red B		3, 7
51 Federal Smoke Red D		7
52 4-Hydroxy-3-(2,4-dimethoxy phenylazo)-1-naphthalene sulfonic acid		1
53 4-Hydroxy-3-(p-methoxyphenylazo)-1-naphthalene sulfonic acid		1
54 4-Hydroxy-3-(2-hydroxy-m-tolylazo)-1-naphthalene sulfonic acid		1
55 4-Hydroxy-3-(o-methoxy-p-chlorophenylazo)-1-naphthalene sulfonic acid		1
56 4-Hydroxy-3-(o-methoxyphenylazo)-1-naphthalene sulfonic acid	Azo Eosine G CI 114	1
57 2-(2-Hydroxy-1-naphthylazo)-1-naphthalene sulfonic acid	Lithol Red R, CI 189 *CI 15630 (CI Pigment Red 49) <sup>8</sup>	1
58 4-Hydroxy-3-phenylazo-1-naphthalene sulfonic acid		1
59 1-(2-Hydroxy-m-tolylazo)-2-naphthol	Oil Bronze	1
60. Indophenol BG		1
61 Indophenol RL		1
62 Induline Base 5G	Mixture of 2,3,7-trianilino-5-phenylphenazinium chloride and 2,3,7,8-tetraanilino-5-phenylphenazinium chloride CI 860	1
63 Isorosinduline		3
64 K1840 (see references 3 and 14)	Ammonium salt of Lithol Red Manufactured by Krebs	7
65 Lithol Red Toner	Manufactured by United	7
66 1-(4-Methoxy anilino) phenylazo)-2-naphthol	Varamine Blue BD Made by General Dyestuff Corp	1
67. 1-(2-Methoxy-4-nitrophenylazo)-2-naphthol	Naphthol Pink, CI 117	1
68 o-Methoxyphenylazo-β-naphthol	Sudan R, Federal Signal Red A, Brilliant Fast Scarlet, Calco Oil Scarlet OBN, MIL-D-3179, CI 113 CI 12150 (Solvent Red 1) <sup>8</sup> CI 12155 (Solvent Red 17) <sup>8</sup>	1, 2, 3, 5, 6, 9, 12
69 5-Methyl-2-anisole-azo-β-naphthol		2
70 4,4'-Methyl-6,6'-chlorothiondigo	Sulfanthrene Pink FF Made by DuPont *CI 73360 (Vat Red 1) <sup>8</sup>	1
71 2-(Monobromo-2-oxo-1(2)-acenaphthylidene-3(2)-thianaphthenone	Ciba Red R, CI 1229 *CI 73865 (Vat Dye) <sup>8</sup>	1
72. α-Naphthylamine-β-naphthol	Federal Red, Naphthylamine Bordeaux *CI 12070 (CI Pigment Red 1)	3, 4, 6, 8
73 β-Naphthylamine-β-naphthol	Azo Turkish Red	3, 4, 6
74 1-(1-Naphthylazo)-2-naphthol	Oil Red, CI 82	1
75 1-(2-Naphthylazo)-2-naphthol	Sudan CB, CI 93	1
76 1-Nitro-acridone		1, 3
77. 1-(2-Nitro-o-tolylazo)-2-naphthol	Toluidine Toner, Lithol Fast Scarlet, CI 69	1, 4
78 2-(2-Oxo-1(2)-acenaphthylidene)-3(2)-thianaphthenone	Sulfanthrene Scarlet Y, CI 1228 *CI 73860 (Vat Red 45)	1
79. Parantroaniline		1, 3, 13
80. Parantroaniline-diethylaniline		3
81. Para Red Toner	Made by United	7
82. Paratoner Red		1, 3, 14
83 Paratoner RT 435D		1

\* See Footnote at end of table



Table 1. (Contd)

Dye		Reference
84. 1-(4-Phenethyl phenylazo)-2-naphthol		1
85. p-Phenetidine-1-hydroxy naphthalene-4-sulfonic acid		3
86. p-Phenetidine- $\beta$ -naphthol		3, 6
87. p-Phenylazo aniline hydrochloride		1
88. 4-Phenylazo-3-hydroxy-2-naphthoic acid	Calcolake Brilliant Red R	1
89. 1-(4-Phenylazo phenylazo)-2-naphthol	Sudan III, Oil Red, CI 248	1, 4
90. Pyronine G	*CI 45005 (Basic Dye) <sup>8</sup>	4
91. Rhodamine	Rhodamine B, Rhodame BEx CI 749	1, 4, 14, 6 10, 12, 15
92. Rhodamine 5GDN base		3
93. Rhodamine 6GDNEx		3, 4, 10
94. Rosinduline		3
95. Rosundone	True Rosindone 7-Phenyl-5-benzo (a) phenazinone	1, 3, 9
96. Safranine T	Mixture of 3,7-diamino-2,8-dimethyl-5-phenyl-phenazinium chloride and 3,7-diamino-2,8-dimethyl-5-O-tolylphenazinium chloride CI 841, Made by DuPont	1
97. Sodium salt of o-carboxylic acid of benzene-azo- $\beta$ -naphthol-3:6-disulphonic acid		3
98. Sodium salt of 4-sulpho-A-naphthalene-azo- $\beta$ -naphthol-6-sulphonic acid		3
99. Sudan IV	Spirit Red III	1, 4, 6, 12
100. Tetrazotized benzidine- $\beta$ -naphthol		6
101. Tetrazotized o-dianisidine- $\alpha$ -naphthol		6
102. Tetrazotized o-tolidine- $\alpha$ -naphthol		6
103. Tetrazotized o-tolidine- $\beta$ -naphthol		6
104. Thioindigo	Durindone Red B, CI 1207 *CI 73300 (Vat Red 41) <sup>8</sup>	1, 6
105. Xylidene- $\beta$ -naphthol		3, 6
106. 1-Xylylazo-2-naphthol	Calco Oil Scarlet II, National Oil Scarlet 6G, Sudan II, Sudan Red *CI 12140 (Solvent Orange 7) <sup>8</sup>	1

\* These are the new Colour Index numbers as best as can be ascertained from the information available.

Table 2. US Food and Drug Administration Dyes

Dye	Colour index number
1. FD&C #40	16035
2. FD&C #3	45430
3. Citrus Red #2	12156
4. FD&C Red #4	14700
5. D&C Red #17	26100
6. D&C Red #31	15800:1
7. D&C Red #34	15880:1
8. D&C Red #39	13058

#### 4. DISCUSSION OF RESULTS

Investigators<sup>1,4,14</sup> generally agree upon the following properties of dyes suitable for the production of colored smokes:

- a. The molecular weight must not exceed 450.
- b. The dye should be a member of one of the following series: anthraquinone, azine, azo, quinoline, or xanthene.
- c. The following groups must be absent: sulfonic, hydrochloride, nitro, nitroso, quaternary ammonium, and oxonium.
- d. The following groups may be present: amino and substituted amino, alkyl, aryl, chloro, bromo, hydroxy, and alkoxy.
- e. The dye must not have a tendency to undergo auto condensation. Of the dyes reviewed, the following have these properties:

- (1) Rhodamine B
- (2) Sudan IV
- (3) 1 Xylylazo-2-naphthol
- (4) Citrus Red #2 (2,5 dimethoxyphenylazo-2-naphthol)
- (5) 9-Diethylaminorosindone
- (6) O-Methoxyphenylazo-B-naphthol

The first four dyes in the list have been shown to be mutagenic or carcinogenic in studies reviewed in the Registry of Toxic Effects of Chemical Substances.<sup>16</sup> In addition, Rhodamine B was listed as a potential health hazard in a report by Anson and Parent,<sup>15</sup> and Citrus Red tends to flame and decompose when disseminated. For these reasons, these dyes were eliminated from further consideration.

The remaining two dyes, 9-diethylaminorosindone and o-methoxyphenylazo- $\beta$ -naphthol, have been recommended by various investigators as replacement dyes for 1-N methylaminoanthraquinone.

#### 4.1 9-Diethylaminorosindone.

Until 1960, 9-diethylaminorosindone, a member of the azine class of dyes was one of the dyes specified for use in the M18 colored-smoke hand grenade. The military specification for this dye was MIL-D-3614<sup>19</sup> which replaced Army 96-111-100. According to Munro and Guion,<sup>9</sup> 9-diethylaminorosindone produced a red smoke comparable in quality and color to that produced by 1-N methylaminoanthraquinone. Difficulties in obtaining the necessary starting materials and the difficult synthesis for the rosindone dyes as compared to the anthraquinone dyes led to the phasing out of rosindone dyes in favor of the anthraquinone dyes by the dye industry.

No present commercial supplier for 9-diethylaminorosindone was located during this study; however, a former manufacturer, Buffalo Color and Chemical Corporation (National Aniline), located in their files a procedure for synthesizing the dye. However, interest in resuming manufacture of the dye was not officially expressed by the company.

No toxicological data<sup>15-18</sup> were located for this dye.

#### 4.2 O-Methoxyphenylazo-B-naphthol.

o-Methoxyphenylazo- $\beta$ -naphthol, a member of the azo class of dyes, has also been specified for use in pyrotechnics (MIL-D-3719) and was used during World War II by the Germans and Italians in pyrotechnic formulations.<sup>12</sup>

A thorough study of this dye by Guion<sup>2</sup> recommended that the dye be used as a replacement only if 1-N-methylaminoanthraquinone or 9-diethylaminorosindone could not be used. The smoke produced by this dye was considered by Guion<sup>2</sup> not of the same quality or hue as that produced by 1-N methylaminoanthraquinone. In addition, the O-methoxyphenylazo-B-naphthol had a tendency to flame more readily when disseminated.

A commercial supplier for this dye, was located during the study. BASF Wyandotte Corporation, markets o-methoxyphenylazo- $\beta$ -naphthol under the name of Sudan Red G.

One report<sup>18</sup> listed this dye as carcinogen, based on unpublished data which could not be corroborated. No other toxicological data were found for this dye.

### 5. CONCLUSIONS AND RECOMMENDATIONS

Two of the 114 dyes reviewed for this study are recommended as candidates for replacement of 1-N methylaminoanthraquinone. The dyes are 9-diethylaminorosindone and o-methoxyphenylazo- $\beta$ -naphthol.

These dyes have been tested and used in pyrotechnics and produce acceptable red smoke. There are insufficient toxicological data at this time to assess the health effects of these dyes.

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ATTN: SGRD-UBD-AL

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Fort Detrick, Bldg 568

Frederick, MD 21701

Headquarters

US Army Medical Research and  
Development Command

ATTN: SGRD-PL

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Fort Detrick, MD 21701

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USA Medical Research Institute of  
Chemical Defense

ATTN: SGRD-UV-L

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Superintendent

Academy of Health Sciences

US Army

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ATTN: HSA-IPM

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Fort Sam Houston, TX 78234

US ARMY MATERIEL DEVELOPMENT AND  
READINESS COMMAND

Commander

US Army Materiel Development and  
Readiness Command

ATTN: DRCLDC

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ATTN: DRCSF-P

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5001 Eisenhower Ave

Alexandria, VA 22333

Project Manager Smoke/Obscurants

ATTN: DRCPM-SMK

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US Army Foreign Science & Technology Center

ATTN: DRXST-MT3

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220 Seventh St., NE

Charlottesville, VA 22901

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US Army Materiel Systems Analysis Activity

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ATTN: DRXSY-TN (Mr. Metz)

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ATTN: DRSMI-RPR (Documents)

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Redstone Arsenal, AL 35809

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DARCOM Field Safety Activity

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ATTN: DRDNA-VT

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Natick, MA 01760

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ATTN: DRDAR-LCU

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ATTN: DRDAR-LCU-CE

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Dover, NJ 07801

Director

Ballistic Research Laboratory

ARRADCOM

ATTN: DRDAR-TSB-S

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US ARMY ARMAMENT MATERIEL READINESS  
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Commander  
US Army Armament Materiel  
Readiness Command

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ATTN: DRSAR-SF  
Rock Island, IL 61299

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ATTN: Technical Library Docu Sect  
Dugway, UT 84022

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US Army Infantry School  
ATTN: NBC Division  
Fort Benning, GA 31905

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Fort McClellan, AL 36205

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US Army Infantry Center  
ATTN: ATSH-CD-MS-C  
Fort Benning, GA 31905

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US Army Infantry Center  
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ATTN: ATZB-DPT-PO-NBC  
Fort Benning, GA 31905

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USA Training and Doctrine Command  
ATTN: ATCD-Z  
Fort Monroe, VA 23651

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USA Combined Arms Center and  
Fort Leavenworth  
ATTN: ATZL-CA-COG  
ATTN: ATZL-CAM-IM  
Fort Leavenworth, KS 66027

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US Army TRADOC System Analysis Activity  
ATTN: ATAA-SL  
White Sands Missile Range, NM 88002

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US Army Test & Evaluation Command  
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ATTN: Army Chemical Officer (Code AC-3)  
Indian Head, MD 20640

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ATTN: Technical Library (Code 343)  
China Lake, CA 93555

Commander Officer  
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Crane, IN 47522

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Director, Development Center  
Marine Corps Development and  
Education Command  
ATTN: Fire Power Division  
Quantico, VA 22134

DEPARTMENT OF THE AIR FORCE

HQ Foreign Technology Division (AFSC)  
ATTN: TQTR  
Wright-Patterson AFB, OH 45433

HQ AFLO/LOWMM  
Wright-Patterson AFB, OH 45433

OUTSIDE AGENCIES

Battelle, Columbus Laboratories  
ATTN: TACTEC  
505 King Avenue  
Columbus, OH 43201

Toxicology Information Center, WG 1008  
National Research Council  
2101 Constitution Ave., NW  
Washington, DC 20418

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ADDITIONAL ADDRESSEE

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US Army Environmental Hygiene Agency  
ATTN: Librarian, Bldg 2100  
Aberdeen Proving Ground, MD 21010

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Bldg. 2340  
Fort Sam Houston, TX 78234

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